

# Exhibit 14.11

United States' Motion to Enter Consent Decree,  
*United States v. Alden Leeds, Inc. et al.*, Civil Action No. 22-7326 (D.N.J.)

# EXHIBIT A-40

**Appendix A** to OxyChem's Comments in Opposition to Proposed Consent Decree,  
*United States v. Alden Leeds, Inc., et al.*, Civil Action No. 2:22-cv-07326 (D.N.J.)

*N.C. Grebe*  
*November 18, 1941*

G-11 Process

(Sodium Salt Method)

Building 36-D.

After the condensation of 500 lbs. of Dowicide #2 with for-  
 maldehyde, 1,000 lbs. of ice are added to the 800<sup>gallon</sup> steel reactor  
 (A) as is the present procedure. The strong acid slurry is then  
 pumped into the existing 600 and 800 gallon lead-lined tanks (B)  
 and (C) which have been previously charged with 400 and 530 gallons  
 of water respectively. The diluted slurry is pumped by means of  
 the same Duriron pump to a 60" acid resistant centrifuge (1). The  
 acid filtrate containing 26% sulfuric acid and wash are sent to  
 the sewer. The wet crude G-11 cake is discharged through the bot-  
 tom of the centrifuge into a cart and is transferred to building  
 47. On the basis of experiments carried out in a 12" centrifuge  
 (RPM 1,500), the wet cake which contains 45% water at this stage  
 has a volume of 19 cubic feet and weighs 910 lbs. Thus, on the  
 basis of a 2" cake in the centrifuge, 4 loads will be necessary.

Building 47.

The wet G-11 cake is hoisted to a platform and dumped into  
 the 1,000 gallon steel tank (2) in which 750 gallons of water and  
 110 lbs. of caustic soda flakes have been previously heated to  
 about 80° C by means of live steam. The temperature is then  
 raised to the boiling point by an additional amount of live steam  
 in order to obtain complete solution of the G-11. The steam in-  
 let should be equipped with an appropriate silencer.

- 1.
- 2.
- 3.

X. Dr. N. Grebe - 10/2/41  
 X. Dr. Grebe - 10/2/41  
 X. Dr. Amell (General) - 12/1/62  
 H. Brandman - 12/1/77  
 J. Virgilio - 3/21/78

2.

Several pounds of Super-Cel are thrown into the hot alkaline solution. The solution is then pumped through a small washing type filter press, (4) (which has been previously heated to about 95° C), in order to remove dirt and a small amount of caustic insoluble resin. The filter should have a capacity of approximately 1 cubic foot and ample filtering area so as to permit filtration to proceed at the rate of 50 gallons per minute. (On the basis of some laboratory experiments the actual volume of cake should be only  $\frac{1}{2}$  cubic foot but for possible variations in the plant a volume of 1 cubic foot is suggested). The pipe lines to and from the filter press are wrapped with a copper steam coil. In addition, the live steam line connected to the bottom outlet of tank (2) is used to blow out the pipe line in either direction. The solution must be maintained above 90° C in order to prevent the precipitation of the G-11 sodium salt. If necessary, a small amount of live steam can be bled into the pump line by means of the above-mentioned steam line. (The solubility of the sodium salt can be increased by the use of an additional amount of caustic soda). The filtrate is returned to tank (2) until it becomes clear and it is then run into the 1,200 gallon steel kettle (5) which has been previously charged with 100 gallons of water.

The filter press is given a hot water wash and is blown with air. This wash water is also sent to tank (5). The press is dumped when time permits.

The pH of the hot alkaline solution is reduced to about 10.5 by means of the slow addition of approximately 95 pounds of 62% sulfuric acid from the lead-lined acid egg (8). The small lead-lined acid tank in building 45 which is no longer required for the Cuminaldehyde

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## 3.

process can be used for this purpose. The sodium salt begins to precipitate when approximately one-half of the required amount of sulfuric acid has been added. The sodium salt precipitates in the form of long fibrous crystals which mat readily and make the slurry very thick. Therefore, very efficient and thorough agitation must be employed during the addition of the acid in order to prevent the precipitation of impurities at a lower pH which would exist in "patches" throughout the slurry if agitation is not thorough. Such impurities would be slow to redissolve on continued stirring. The acid inlet should be installed so that the acid does not run down the wall of the tank but falls clear of the sides.

The slurry is cooled to room temperature by circulating cold water through the jacket. (Steam should also be connected to this jacket in case it ever becomes necessary to heat the solution before precipitation as may be necessary after a shut down). A close fitting agitator is required for this cooling process as the sodium salt will deposit on the cooling surface and considerably retard the rate of cooling. This deposit is very soft and therefore a scraping type agitator is not necessary. (Sometimes this sodium salt deposit builds up to a thickness of more than 2" on the walls of the crystallizer located in 47 building. This crystallizer is equipped with a turbine type agitator).

After cooling to room temperature, the slurry is pumped to a 48" centrifuge (7). The pump must not be a close clearance pump as the G-11 sodium salt will not pass through the screen of a "Westco" type pump. Based on experiments in the 12" centrifuge, the G-11

4.

sodium salt cake weighs approximately 790 pounds, contains 50% moisture and has a density of 60 pounds per cubic ft. Thus, for a 2" cake, 4 loads will be necessary in a 48" centrifuge. The pH of the filtrate is 10.5 and contains sodium sulfate and the sodium salts of Dowicide and resinous by-products. A bronze centrifuge basket will probably be suitable, however, the possibility of contaminating the G-11 sodium salt with traces of copper should be considered, as traces of copper are known to cause discoloration of soap.

790 / 60 = 13.2

In this process, no attempt is made to recover the Dowicide in the filtrate and therefore the filtrate is discharged to the sewer. More experiments will be made in the laboratory in order to determine if the Dowicide content can be definitely reduced to such a value that it will not be economical to recover it from the filtrate and at the same time obtain a satisfactory yield. If the Dowicide must be recovered, additional equipment, i.e., a 1,200 gal. lead-lined tank, pump and vacuum still will be required.

The G-11 sodium cake after washing with water in the centrifuge is discharged through the bottom of the centrifuge. The cake is hoisted to a platform and dumped into the 1,000 gallon lead-lined(9) steel tank which has been previously charged with 800 gallons of water. The slurry is made acid to Congo Red by means of the addition of approximately 80 lbs. of 62% sulfuric acid in order to decompose the sodium salt and obtain the free acid. Agitation in this tank must also be thorough as the slurry is quite thick. In addition, the agitator must be capable of breaking up the G-11 sodium salt cake so that the sulfuric acid can react with the individual particles and thus insure the complete conversion of the sodium salt to the free acid.

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5.

Sodium bicarbonate is then added in order to raise the pH to approximately 7.5 and the G-11 slurry is then pumped to a 48" centrifuge equipped with a bronze basket. The pump (10) should be similar to pump (6) as the G-11 clogs the strainers of Westco pumps. Based on a 2" cake approximately  $5\frac{1}{2}$  loads will be required to filter the batch. (Wt. wet cake - 680 lbs.; moisture 45%; density 42 lbs./1 cu. ft.). In view of the fact that more than 4 loads will be required to filter the batch, it may be advisable to use a 60" centrifuge. Experiments will be made in the laboratory to determine if the G-11 can be precipitated in a denser form and thus be able to filter the batch in less than 5 loads in a 48" centrifuge (cake thickness 2").

The filtrates from the centrifuges should run first to 200 gallon "catch all" tanks (separate one for each centrifuge) before flowing to the sewer so that in case the filter medium breaks or the filtrate does not run clear, no G-11 will be lost. The centrifuge pumps should be connected so that the cloudy filtrate in the "catch all" tank can be recirculated through the centrifuge.

After washing with water, the G-11 cake is dried in the existing vacuum shelf drier.

#### Capacity of Equipment.

With the exception of reactor (A), the above equipment should be capable of processing 3 batches (375 lbs. G-11 Refined per batch) or approximately 1,000 pounds per twenty-four hour day. In order to attain this production rate, at least 2 more or one larger reactor must be installed.

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6.

If centrifuges cannot be obtained, consideration should be given to other types of filtration equipment such as a Vallez Filter which could replace either of the last two centrifuges (7) or (11), or a Sweetland Filter which could replace the centrifuge (7) in which the sodium salt is filtered.

H. G. Krebs

Delawanna, N. J.

November 18, 1941

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